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(54) SURFACE-TREATED CALCIUM CARBONATE PRODUCT AND PRODUCTION METHOD THEREFOR

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a product containing a surface-treated calcium carbonate powder having low water content.

SOLUTION: This product is prepared by sealing a composition prepared by homogeneously mixing a surface-treated calcium carbonate powder with a dehydrating agent powder into a bag formed from an airtight material. Water contained in the calcium carbonate powder is removed by the dehydrating agent powder; therefore, the calcium carbonate powder is dried merely by allowing the product to stand for a suitable time. This product is obtained by subjecting a calcium carbonate powder to a surface treatment, drying the powder, mixing it with a dehydrating agent powder, and sealing the mixture into a bag formed from an airtight material. When the calcium carbonate powder in this product is compounded as a thixotropic agent or a filler into a one-component moisture-curable resin composition, the resin composition is prevented from curing during its storage because of the low water content of the calcium carbonate powder.

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CLAIMS

[Claim(s)]

[Claim 1]Surface treatment calcium carbonate products which enclose a constituent which mixes uniformly surface treatment calcium carbonate powder and dehydrator powder with a bag which comprised an airtight raw material, and are characterized by things.

[Claim 2]A manufacturing method of surface treatment calcium carbonate products enclosing with a bag which performed a surface treatment to calcium carbonate powder, obtained surface treatment calcium carbonate powder, was subsequently promptly mixed with dehydrator powder after desiccation, and comprised an airtight raw material.

[Claim 3]A drying method of surface treatment calcium carbonate powder mixing uniformly surface treatment calcium carbonate powder and dehydrator powder, and enclosing with a bag which comprised an airtight raw material.

[Claim 4]A liquid moisture curing type resin composition which opens the surface treatment calcium carbonate product according to claim 1, and blends taken-out surface treatment calcium carbonate powder.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[001] [Field of the Invention] This invention relates to the surface treatment calcium carbonate powder

blended as a thixotropy agent or a bulking agent into the resin composition used mainly as adhesives, a sealing material, etc.

[002] [Description of the Prior Art] In many cases, the thixotropy agent or the bulking agent is blended into the adhesives used in the civil-engineering and construction field etc., or a sealing material. This is for the field where adhesives etc. are applied not being the level surface in many cases, and mainly preventing lappets, such as adhesives. As a thixotropy agent or a bulking agent, although colloidal silica, calcium carbonate powder, etc. are used, surface treatment calcium carbonate powder is convenient in recent years.

[003] Surface treatment calcium carbonate powder processes the calcium-carbonate-powder surface with fatty acid ester, fatty acid salt, etc., and does not condense it easily in the resin composition used as adhesives etc. and it is easy to distribute it uniformly. Therefore, without checking the adhesive property of adhesives etc., it is fond as what gives good thixotropy, and is briskly blended with the 1 liquid moisture curing type resin composition or 2 liquid mixing hardening resin constituent used as adhesives etc.

[004] However, when surface treatment calcium carbonate powder was blended with a 2 liquid mixing hardening resin constituent, a problem was hardly produced, but when blending into a 1 liquid moisture curing type resin composition, there were the following problems. That is, when surface treatment calcium carbonate powder was blended into the 1 liquid moisture curing type resin composition, the problem of having thickened or gelling was during storage. As for this, surface treatment calcium carbonate powder is because not less than 2000 ppm of moisture is contained in addition in many cases and a 1 liquid moisture curing type resin composition fully hardens the usual desiccation with this moisture as a line.

[005] For this reason, when surface treatment calcium carbonate powder was blended into a 1 liquid moisture curing type resin composition, blending by azeotropy, scale loss pressure, or other means, after carrying out dehydrating treatment was performed. After blending, drying the whole constituent is also performed by scale loss pressure or other means. However, any means needed special equipment of azeotropy equipment, scale loss pressure equipment, etc., and had the grudge that a 1 liquid moisture curing type resin composition could not be manufactured rationally.

[006] [Problem(s) to be Solved by the Invention] Then, the grade from which, as for this invention persons, a 1 liquid moisture curing type resin composition does not start hardening for the moisture content of surface treatment calcium carbonate powder. Namely, in order to make 1500 ppm or less reduce moisture content at least. When many things were examined, surface treatment calcium carbonate powder was mixed with dehydrator powder to homogeneity, it enclosed with the airtight bag and a certain amount of thing for which moisture content can be reduced to 1500 ppm or less if time neglect is carried out was discovered not to expect. This invention is made based on such discovery.

[007] [Means for Solving the Problem] That is, it is related with a surface treatment calcium carbonate product which this invention encloses a constituent which mixes uniformly surface treatment calcium

carbonate powder and dehydrator powder with a bag which comprised an airtight raw material, and is characterized by things, and a manufacturing method for the same.

[008] Surface treatment calcium carbonate powder used by this invention should be conventionally publicly known just. Calcium carbonate powder which is fatty acid ester, fatty acid salt, a sulfate ester type anionic surfactant, a sulfonic acid type anionic surfactant, etc. and by which the surface was specifically processed is used. Particle diameter of surface treatment calcium carbonate powder should just also be a public domain conventionally. Generally, what has proper particle size distribution within the limits of 0.01~30 micrometers is used.

[009] Arbitrary things will be used if it comes conventionally to grind a substance currently used as a dehydrator as dehydrator powder used by this invention. Zeolite powder, silico gel powder, etc. which adsorb water physically, such as calcium oxide powder, metal oxide powder, etc. which specifically adsorb water chemically, are used. Although particle diameter of dehydrator powder is also arbitrary, in order to make it mix with surface treatment calcium carbonate powder as uniformly as possible, a thing with particle size distribution comparable as surface treatment calcium carbonate powder is preferred. However, when it is necessary to sort out dehydrator powder and surface treatment calcium carbonate powder later. That is, when both blend surface treatment calcium carbonate powder and dehydrator powder with moisture curing type resin and there is a possibility that moisture curing type resin may harden with moisture in dehydrator powder, a thing with completely different particle size distribution is preferred. It is because a means of sieving can sort out both if it has different particle size distribution.

[010] As for the mixing ratio of surface treatment calcium carbonate powder and dehydrator powder, it is preferred that dehydrator powder is one to 30 weight section to surface treatment calcium carbonate powder 100 weight section. It is more preferred especially that it is five to 15 weight section, and it is most preferred that it is six to 10 weight section. If dehydrator powder will be less than one weight section, a tendency to become difficult to reduce moisture content of surface treatment calcium carbonate powder to 1500 ppm or less will arise. If dehydrator powder exceeds 30 weight sections, it may have an adverse effect on the characteristic of a moisture curing type resin composition obtained. Here, moisture content is measured and computed by the following methods. That is, when weight of surface treatment calcium carbonate powder which it is going to measure is set to w_0 , azeotropy of this is carried out and it asks for weight w_0 of an absolute dry condition, $[(w_0 - w_0') / w_0'] \times 100$. Surface treatment calcium carbonate powder and dehydrator powder do not interfere, even if it mixes at which time, but immediately after most desirable one manufactures surface treatment calcium carbonate powder, they are. That is, a surface treatment is performed to calcium carbonate, surface treatment calcium carbonate powder is obtained, and, subsequently mixing with dehydrator powder promptly is most preferred after desiccation. It is at this time immediately after drying surface treatment calcium carbonate powder, and is because moisture content has decreased most.

[011] A constituent with which surface treatment calcium carbonate powder and dehydrator powder were mixed uniformly is enclosed with a bag which comprised an airtight raw material. As an airtight raw material, water may be a grade which does not invade easily and specifically. A layered product of a deposition film, a synthetic resin film, and paper which vapor-deposited metal, such as aluminum, kraft, etc. are used for synthetic resin films, such as metallic foils, such as aluminum foil, and a polyethylene film, and a synthetic resin film. What is necessary is to bend or pile up an airtight raw material and just to sew the circumference on, in order to form a bag for an airtight raw material. And if a mouth in a bag is bound with a string or elastic yarn to it after throwing a constituent into a mouth in a bag, it can enclose with it easily. Although there is invasion about some steam from a binding part of the surrounding portion being attached by sewing or a mouth in a bag, invasion of such a steam is a grade which does not interfere in this invention.

[012] After surface treatment calcium carbonate products enclosed as mentioned above are kept in a warehouse, they are conveyed with *** and a consumer is provided with them. And it is kept by the consumer side, it will open if needed, and will be used for manufacture of a liquid moisture curing type resin composition etc. Therefore, after surface treatment calcium carbonate products are manufactured, by the time it uses it by the consumer side, two days thru/ or several months will be required at least. Moisture which dehydrator powder contains in surface treatment calcium carbonate powder is absorbed, and moisture content of surface treatment calcium carbonate powder decreases

in this period. That is, a period until it uses it from a manufacturer of surface treatment calcium carbonate powder, being provided for the consumer's side will say that desiccation of surface treatment calcium carbonate powder is presented. Therefore, this invention can mix uniformly surface treatment calcium carbonate powder and dehydrator powder, and can also grasp them as a drying method of surface treatment calcium carbonate powder of enclosing with a bag which comprised an airtight raw material.

[0013] surface treatment calcium carbonate products concerning this invention with which a consumer was provided are opened — dehydrator powder — or after sorting out dehydrator powder, surface treatment calcium carbonate powder is blended with moisture curing type resin, and a 1 liquid moisture curing type resin composition is manufactured. What is necessary is for dehydrator powder to adsorb moisture chemically at the inside, and to blend surface treatment calcium carbonate powder with dehydrator powder, and just to manufacture a 1 liquid moisture curing type resin composition, when there is no possibility of stiffening moisture curing type resin. What is necessary is to sort out dehydrator powder, to blend only surface treatment calcium carbonate powder, and just to manufacture a 1 liquid moisture curing type resin composition, when there is a possibility that moisture with which dehydrator powder was adsorbed may stiffen moisture curing type resin. About 50-1000 weight sections may be sufficient as a blending ratio of surface treatment calcium carbonate powder to inside of a 1 liquid moisture curing type resin composition to moisture curing type resin 100 weight section. Surface treatment calcium carbonate powder in a product concerning this invention, since it is fully dried and moisture content has become 1500 ppm or less with a dehydrator, even if it blends into a 1 liquid moisture curing type resin composition, it is hard to harden during storage, therefore thickening and gelling of a 1 liquid moisture curing type resin composition can be prevented. As a 1 liquid moisture curing type resin composition, a thing containing modified silicone resin, polyurethane resin, etc. is used, and it is used as adhesives, a sealing material, or a paint in various fields, such as civil engineering and construction, a car, and electronic equipment. Although a 1 liquid moisture curing type resin composition was mainly explained above, surface treatment calcium carbonate powder in surface treatment calcium carbonate products concerning this invention can be used for a 2 liquid hardening resin constituent etc. as a bulking agent as a

[0014] [Example] Hereafter, although this invention is explained based on an example, this invention is not limited to an example. If this invention mixes surface treatment calcium carbonate powder with dehydrator powder to homogeneity and it encloses with the airtight bag, Even when the moisture content of surface treatment calcium carbonate powder can be reduced to 1500 ppm or less and it blends with moisture curing type resin, I should be understood as a thing based on discovery that hardening of moisture curing type resin under storage can be prevented.

[0015] [Example 1] — the fatty acid system surface treatment calcium carbonate powder by Konoshima Chemical, Inc. (trade name: calces PL-S505) was prepared first. That mean particle thixotropy agent.

diameter of this surface treatment calcium carbonate powder is 0.1 micrometer. Moisture content was a 3000 ppm thing.

After mixing uniformly calcium-oxide-powder (mean particle diameter is about 0.1 micrometer) 7 weight section as dehydrator powder to this surface treatment calcium-carbonate-powder 100 weight section, it stored into the bag formed with aluminum foil, the mouth was heat sealed and sealed, and surface treatment calcium carbonate products were obtained. And when this is neglected for one month, it means that the moisture content of surface treatment calcium carbonate powder is 800 ppm, and calcium oxide powder was adsorbed in the moisture equivalent to 2200 ppm. [016] The surface treatment calcium carbonate powder (dehydrator powder is included,) after the above-mentioned one-month neglect was taken out, and the following 1 liquid moisture curing type resin compositions were manufactured using this.

SUMIDULE E21-2 100 weight section. SUMIDULE E21-2 100 weight section Surface treatment calcium carbonate powder 107 weight sections diethyl phthalate — SUMIDULE E21-2 is a urethane prepolymer by Sumitomo BAYER urethane incorporated company 30 weight section here.

The content of an end isocyanate group (NCO) is 9.0 % of the weight, and viscosity is 4 Pa-s (23 **). SUMIDULE E21-2 100 weight section. SUMIDULE E21-2 100 weight section Surface treatment calcium carbonate powder 107 weight sections diethyl phthalate — SUMIDULE E21-2 is a urethane prepolymer by Sumitomo BAYER urethane incorporated company 30 weight section here.

The content of an end isocyanate group (NCO) is 9.0 % of the weight, and viscosity is 4 Pa-s (23 **). Sankyo BL is a tin catalyst by Sankyo Organic Chemicals, Inc.

It is a polymerization catalyst of a urethane prepolymer.

[0017] It is permurite powder (with a potassium sodium aluminosilicate type,) as dehydrator powder to surface treatment calcium-carbonate-powder 100 weight section used in example 2 Example 1. Mean particle diameter is 3 A. After mixing seven weight sections uniformly, it stored into the bag formed with aluminium foil, the moth in a bag was bound with a string, and surface treatment calcium carbonate products were obtained. And when this is neglected for one month, it means that the moisture content of surface treatment calcium carbonate powder is 900 ppm, and calcium oxide powder was adsorbed in the moisture equivalent to 21.00 ppm. The surface treatment calcium carbonate powder (dehydrator powder is included,) after the above-mentioned one-month neglect was taken out, and the 1 liquid moisture curing type resin composition of the same presentation as Example 1 was manufactured using this.

[0018] the 1 liquid moisture curing type resin composition of the same presentation as Example 1 was manufactured without mixing with dehydrator powder namely, -- using processing calcium-carbonate-powder (calcios PL-S305) 107 weight section used in comparative example 1 Example 1 as

[0019]Processing calcium-carbonate-powder (calcites PLS505) 100 weight section used in comparative example 2 Example 1, Calcium-oxide-powder 7 weight section used in Example 1 was mixed at the time of manufacture of a 1 liquid moisture curing type resin composition, without mixing with the bag made from aluminium foil, and also the 1 liquid moisture curing type resin composition of the same presentation as Example 1 was manufactured.

[0020]Restoration sealing of the viscosity of the 1 liquid moisture curing type resin composition obtained by Examples 1 and 2 and the comparative examples 1 and 2 and this 1 liquid moisture curing type resin composition was carried out by the aluminium bag, and when the viscosity after carrying out prescribed period storage at ordinary temperature was measured, the result shown in Table 1 was obtained. Therefore, the 1 liquid moisture curing type resin composition concerning Examples 1 and 2 is understood that storage stability is good compared with the thing concerning the comparative examples 1 and 2. Viscosity was measured under 23 °C conditions.

[0021]  Table 1

[0021] Table 11

Learner		新規例1		新規例2		比較例1		比較例2	
スマジュー	ル E 2 1 - 2	1 0 0	1 0 0	1 0 0	1 0 0	1 0 0	1 0 0	1 0 0	1 0 0
スタン	B L	0 . 0 2	0 . 0 2	0 . 0 2	0 . 0 2	0 . 0 2	0 . 0 2	0 . 0 2	0 . 0 2
表面選択的	機能化シマム期末 (表面候補候補を含む。)	1 0 7	1 0 7						
表面選択的	機能化シマム期末 (表面候補候補を含む。)			1 0 7	1 0 7	1 0 0	1 0 0	1 0 0	1 0 0
選化	シマム期末					1 0 7	1 0 7	1 0 0	1 0 0
フタル	ジオチル	3 0	3 0	3 0	3 0	3 0	3 0	3 0	3 0
粘膜 (<i>Pa-s</i>) うちの条件で 満足	無選擇	1 6 0	1 7 0	4 5 0	2 6 0				
	1回後	1 6 0	1 7 5	ダル化	7 6 0				
	1ヶ月後	1 7 5	1 9 0						
	3ヶ月後	1 8 0	1 9 0						

[0022] The mixing ratio of example 3 calcium oxide powder was made into five weight sections to surface treatment calcium-carbonate-powder 100 weight section, and also surface treatment calcium carbonate products were obtained like Example 1, and the 1 liquid moisture curing type resin composition was obtained like Example 1. When this storage stability was evaluated like Example 1, the almost same result was accepted.

[0023] The mixing ratio of example 4 calcium oxide powder was made into three weight sections to surface treatment calcium-carbonate-powder 100 weight section, and also surface treatment calcium carbonate products were obtained like Example 1, and the 1 liquid moisture curing type resin composition was obtained like Example 1. When this storage stability was evaluated like Example 1,

the almost same result was accepted.

[0024]The surface treatment calcium carbonate powder (dehydrator powder is included,) after one-month neglect obtained in example 5 Example 2 was taken out, and the 1 liquid moisture curing type resin composition of the following presentation was manufactured using this.

Epoxy resin 100 weight section Hardening agent 40 weight section for epoxy resins Modified silicone resin 100 weight section Catalyst for modified silicone resin The amount part of duplexes Surface treatment calcium carbonate powder 300 weight section here, As an epoxy resin, trade name Epicat 828 made from oil recovery shell epoxy was used. As a hardening agent for epoxy resins, the trade name H-30 made from oil recovery shell epoxy was used. As modified silicone resin, the Kaneka trade name MS polymer 303 was used. As a catalyst for modified silicone resin, the dibutyl tin compound (the Sankyo Organic Chemicals make, trade name Star No 918) was used.

[0025]When the same method as Example 1 estimated the storage stability of this 1 liquid moisture

curing type resin composition, thickening was hardly accepted.

[0026]

[Effect of the Invention]If the surface treatment calcium carbonate products concerning this invention are used, the moisture content of the surface treatment calcium carbonate powder in the product is reduced by below half grade compared with what is usually marketed. Therefore, since it is hard to harden during storage when a 1 liquid moisture curing type resin composition is manufactured using this surface treatment calcium carbonate powder, the effect of excellency in storage stability is done so.

[0027]If the surface treatment calcium carbonate products concerning this invention are used, the process, for example, an azeotropy process etc., of drying moisture from surface treatment calcium carbonate powder will become unnecessary. Therefore, the effect that manufacture of a 1 liquid moisture curing type resin composition can be rationalized also does so.

[0028]Above, although the case where the surface treatment calcium carbonate products concerning this invention were used when manufacturing a 1 liquid moisture curing type resin composition was explained, also when manufacturing a 2 liquid mixing hardening resin constituent or other various constituents, it can mainly use as a thixotropy agent or a bulking agent.

[Translation done.]